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# Environmental Technology Verification Program

## Advanced Monitoring Systems Center

Generic Verification Protocol for Technologies for Rapid Detection of Soil Toxicity

ETV ETV ETV

# Generic Verification Protocol

**for**

# Verification of
# Technologies for Rapid Detection of Soil Toxicity

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# DISTRIBUTION LIST

**U.S. Environmental Protection Agency**

Advanced Monitoring Systems (AMS) Center Project Officer
AMS Quality Manager

**Battelle AMS Center**

AMS Center Manager
Verification Testing Leader
Verification Test Coordinator
Quality Manager
Technical Staff

**Peer Reviewers**

**Vendors of Technologies for Rapid Detection of Soil Toxicity**

**Reference Laboratory**

**Test Facility, if applicable**

**Test Collaborators, if applicable**

**Subcontractors, if applicable**

## LIST OF ABBREVIATIONS/ACRONYMS

| | |
|---|---|
| AMS | Advanced Monitoring Systems |
| COA | certificate of analysis |
| COC | chain-of-custody |
| DQI | data quality indicator |
| $EC_{50}$ | median effective concentration causing 50% inhibition |
| EPA | U.S. Environmental Protection Agency |
| ETV | Environmental Technology Verification |
| LRB | laboratory record book |
| NIST | National Institute of Standards and Technology |
| PCB | polychlorinated biphenyl |
| PD | percent difference |
| pdf | Adobe portable document format |
| PE | performance evaluation |
| QA | quality assurance |
| QC | quality control |
| QCS | quality control samples |
| QMP | quality management plan |
| RSD | relative standard deviation |
| SOP | standard operating procedure |
| TCDD | 2,3,7,8-tetrachlorodibenzodioxin |
| TQAP | test/quality assurance plan |
| TSA | technical systems audit |

# SECTION A
# PROJECT MANAGEMENT

## A1 VERIFICATION TEST ORGANIZATION

This protocol provides generic procedures for implementing a verification test for technologies that rapidly detect soil toxicity. Verification tests are conducted under the auspices of the U.S. Environmental Protection Agency (EPA) through the Environmental Technology Verification (ETV) Program. Verification tests of monitoring technologies are coordinated by Battelle, which manages the ETV Advanced Monitoring Systems (AMS) Center through a cooperative agreement with EPA. The scope of the AMS Center covers verification of monitoring technologies for contaminants and natural species in air, water, and soil. In performing verification tests, Battelle follows the procedures specified in this protocol and complies with quality requirements in the "Quality Management Plan (QMP) for the ETV Advanced Monitoring Systems Center" (AMS Center QMP).(1)

Verification tests are performed by Battelle in cooperation with EPA and the vendors whose technologies for rapid detection of soil toxicity are being verified. These test procedures may be performed by Battelle, test facility staff, a qualified collaborator, and/or a qualified subcontractor. The specific staff members who will perform the test procedures are referred to as "technical staff" in this protocol. Each technology vendor is expected to provide Battelle with their respective technology and will train the technical staff in their technology use.

Quality assurance (QA) oversight will be provided by the Battelle Quality Manager and also by the EPA AMS Center Quality Manager, at his or her discretion. The organization chart in Figure 1 identifies the responsibilities of the organizations and individuals associated with the verification test. Solid lines in the figure indicate direct reporting relationships, dashed lines indicate indirect reporting relationships. Roles and responsibilities are defined further below.

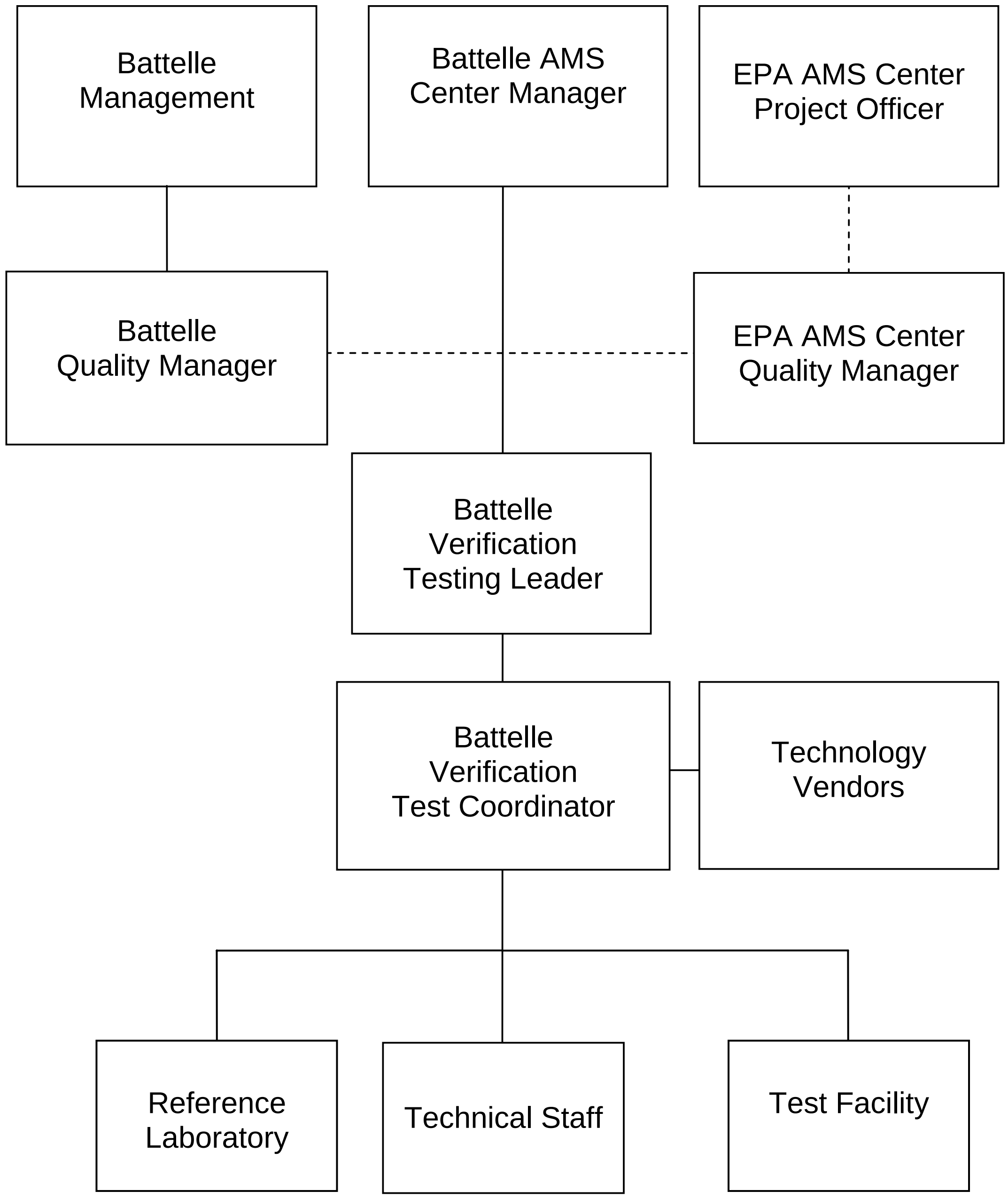


**Figure 1. Organization Chart for the Verification Test**

### A1.1 Battelle

The AMS Center's Verification Test Coordinator has overall responsibility for ensuring that the technical, schedule, and cost goals established for the verification test are met. Specifically, the Verification Test Coordinator will:

- Prepare a draft test/QA plan (TQAP) based on this protocol.
- Prepare draft verification reports and verification statements.
- Establish a budget for the verification test and manage staff to ensure the budget is not exceeded.
- Revise the draft TQAP, verification reports, and verification statements in response to reviewer comments.
- Assemble a team of qualified technical staff to conduct the verification test.
- Direct the team in performing the verification test in accordance with this protocol and any TQAP prepared based on this protocol.
- Hold a kick-off meeting approximately one week prior to the start of the verification test to review the critical logistical, technical, and administrative aspects of the verification test. Responsibility for each aspect of the verification test will be confirmed.
- Ensure that all quality procedures specified in this protocol and in the AMS Center QMP(1) are followed.
- Serve as the primary point of contact for technology vendors.
- Ensure that confidentiality of sensitive vendor information is maintained.
- Assist vendors as needed during verification testing.
- Become familiar with the operation and maintenance of the technologies through instruction by the vendors, if needed.
- Respond to any issues raised in assessment reports, audits, or from test staff observations, and institute corrective action as necessary.
- Coordinate distribution of the final TQAP, verification reports, and verification statements.

The Battelle Verification Testing Leader will provide technical guidance and will:

- Support the Verification Test Coordinator in preparing the TQAP and organizing the testing.
- Review the draft and final TQAP.
- Attend the verification test kick-off meeting.
- Review the draft and final verification reports and verification statements.

The Battelle AMS Center Manager will:

- Review the draft and final TQAP.
- Review the draft and final verification reports and verification statements.
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the verification test.
- Ensure that confidentiality of sensitive vendor information is maintained.
- Support the Verification Test Coordinator in responding to any issues raised in assessment reports and audits.
- Maintain communication with EPA's technical and quality managers.
- Issue a stop work order if Battelle or EPA QA staff discovers adverse findings that will compromise test results.

Battelle's Quality Manager for the AMS Center will:

- Review the draft and final TQAP.
- Attend the verification test kick-off meeting.
- Conduct a technical systems audit (TSA) once during the verification test, or designate other QA staff to conduct the audit.
- Audit at least 10% of the verification data or designate other QA staff to conduct the data audit.
- Prepare and distribute an assessment report for each audit.
- Verify implementation of any necessary corrective action.

- Request that Battelle's AMS Center Manager issue a stop work order if audits indicate that data quality is being compromised.
- Provide a summary of the QA/QC activities and results for the verification reports.
- Review the draft and final verification reports and verification statements.

### A1.2 Technology Vendors

The responsibilities of the technology vendors are as follows:

- Review and provide comments on the draft TQAP.
- Accept (by signature of a company representative) the final TQAP prior to test initiation.
- Provide their technology for evaluation during the verification test.
- Provide all other equipment/supplies/reagents/consumables needed to operate their technology for the duration of the verification test.
- Supply a representative to provide training on their technology use, and provide written consent and instructions for test staff to carry out verification testing, including written instructions for routine operation of their technology.
- Provide maintenance and repair support for their technology, on-site if necessary, throughout the duration of the verification test.
- Review and provide comments on the draft verification report and statement for their respective technology.

### A1.3 EPA

EPA's responsibilities in the AMS Center are based on the requirements stated in the "Environmental Technology Verification Program Quality Management Plan" (EPA ETV QMP).[2] The roles of specific EPA staff are as follows:

The EPA's AMS Center Quality Manager will:

- Review the draft TQAP.
- Perform at his or her option one external TSA during the verification test.

- Notify the EPA AMS Center Project Officer of the need for a stop work order if the external audit indicates that data quality is being compromised.
- Prepare and distribute an assessment report summarizing results of the external audit.
- Review draft verification reports and verification statements.

The EPA's Project Officer for the AMS Center will:

- Review the draft TQAP.
- Approve the final TQAP.
- Notify the Battelle AMS Center Manager of the need for a stop work order if it is discovered that data quality is being compromised.
- Review the draft verification reports and verification statements.
- Oversee the EPA review process for the TQAP, verification reports, and verification statements.
- Coordinate the submission of verification reports and verification statements for final EPA approval.

### A1.4 Technical Staff

The technical staff will support the Verification Test Coordinator in planning and conducting the verification test. The responsibilities of the technical staff will be to:

- Assist in planning for the test, and making arrangements for the receipt of and training on the technologies.
- Attend the verification test kick-off meeting.
- Assist vendor staff as needed during technology receipt and training.
- Conduct verification testing using the vendor's technology, if necessary.
- Conduct reference testing where applicable or help coordinate with laboratories providing reference testing.
- Perform statistical calculations specified in this protocol on the technology data as needed.

- Provide results of statistical calculations and associated discussion for the verification reports as needed.
- Support the Verification Test Coordinator in responding to any issues raised in assessment reports and audits as needed.

### A1.5 Reference Laboratory

When valid reference methods are available, the contaminant concentration of the soil test samples will be confirmed. When the analyses are within Battelle's capabilities, Battelle will perform the analyses; however, if it is more cost effective, Battelle may collaborate with another laboratory or establish a subcontract with a commercial laboratory to perform the analyses.

In order to be selected to perform the reference analyses during the verification test, a commercial laboratory will need to provide Battelle documentation that demonstrates its competence to perform the needed analysis; such documentation may include copies of its method/standard operating procedure (SOP), QA manual, state government certifications/approvals for analysis of the appropriate contaminant, and/or staff training records, where available. If the prospective laboratory does not demonstrate its capability adequately, another laboratory will be selected and its competence verified in a similar manner.

### A1.6 Test Facility

Either Battelle or another appropriate facility such as a laboratory that conducts soil testing will serve as the test facility. The test facility personnel are expected to:

- Identify a point of contact for the test who will serve as the primary interface with the Verification Test Coordinator.
- Attend the verification test kick-off meeting.
- Ensure that test facility staff and facilities are ready for the verification test.
- Assist Battelle's Verification Test Coordinator in ensuring that the verification testing is conducted in accordance with this protocol and any TQAPs that are generated.

- Assist Battelle and technology vendor staff as needed during technology receipt, training, testing, and return of equipment to technology vendors.
- Ensure that necessary test facility resources (e.g., space and power) are committed to the verification test.

## A2 BACKGROUND

The ETV Program's AMS Center conducts third-party performance testing of commercially available technologies that detect or monitor natural species or contaminants in air, water, and soil. The purpose of ETV is to provide objective and quality-assured performance data on environmental technologies, so that users, developers, regulators, and consultants can make informed decisions about purchasing and applying these technologies. Stakeholder committees of buyers and users of such technologies recommend technology categories, and technologies within those categories, as priorities for testing. Technologies for rapidly detecting soil toxicity were identified as a priority technology category through the AMS Center stakeholder process since they have the potential to make the evaluation of soil toxicity more efficient and timely.

Soil toxicity testing can be used at hazardous waste sites to screen for particular areas of concern or to assist in monitoring the effectiveness of cleanup. Soil toxicity tests do not require knowing the contaminants present at the site; they are typically used as a broad range screen of all potentially toxic compounds that may be present. Traditional soil toxicity tests include evaluations such as seed germination and root elongation, as well as organism-based tests such as earthworm survival.[(3)] Tests such as these can take several weeks to achieve results. This protocol provides procedures for a verification test of rapid analysis technologies that detect toxicity in soil. The objective of this soil toxicity technology verification test is to evaluate the technology's ability to detect certain analytes that are particularly toxic to humans by adding them, individually, to a controlled experimental matrix, as well as by testing various "real-world" soil samples where the toxins may be present alone or with various other toxins. This generic protocol outlines testing for a number of contaminants that are common to site cleanups and known to be toxic, but does not include all toxic compounds or testing in all situations which may be encountered in a site cleanup or evaluation situation. Data generated from verification tests based on this generic protocol are intended to provide one set of objective and quality assured performance data on environmental technologies, to assist users, developers, regulators, and consultants in making informed decisions about purchasing and properly applying these technologies.

This verification test will determine the performance characteristics of commercially available technologies that can provide results within a 24-hour period to make soil toxicity determinations more efficient. Critical characteristics of the soil toxicity technologies that will be assessed during this testing include the following:

- Endpoint
- Precision
- False negative rate
- False positive rate
- Lowest detectable level
- Matrix effects
- Data completeness
- Operational factors such as ease of use and maintenance
- Field portability.

## A3 VERIFICATION TEST DESCRIPTION AND SCHEDULE

### A3.1 Summary of Technology Category

Conventional soil toxicity methods often can take weeks to achieve results. Technologies to be evaluated in the verification of rapid soil toxicity technologies include those that produce results within a 24-hour period. Such rapid soil toxicity technologies have the potential to expedite the decision-making process for regulators. Technologies applicable to this technology category can be those designed to directly test the soil, or just the soil extract. Rapid soil toxicity technologies do not provide a measured concentration of specific toxins; rather, they provide a broad range screen of the toxic nature of the soil. For example, a soil extract may be added to a bacteria, bioluminescent plankton, or other such organism or compound producing a measurable response that varies based on the toxicity of contaminants in the soil. The specific response may vary by technology, but could include a change in color or light intensity, respiration rate, or other response that is proportional to the concentration level of the contaminant.

### A3.2 Verification Test Schedule

A verification test following this protocol should take between ten months and one year to complete. Test planning and preparation may take place over a period of four to five months. Actual testing should be completed within one month. Data review and reporting should be completed within four to five months. Table 1 shows a general schedule of testing and data analysis/reporting activities to be conducted in a verification test that follows this protocol. The test procedures are described in Section B of this protocol. Subsequent to testing, a separate verification report will be drafted for each technology. Each draft report will be peer-reviewed, revised, and submitted to EPA for final approval. Technologies for detecting soil toxicity and associated equipment (but not consumables) will be returned to the vendors at the completion of report writing.

### A3.3 Test Facility

The test facility should be a location that can accommodate laboratory testing of technologies for detecting toxicity in soil. This could be laboratory facilities at Battelle or other such laboratory facilities that routinely test soil. Field portability testing, if applicable, will be conducted by transporting the technology from a laboratory to a non-laboratory area. In addition to a traditional field setting, non-laboratory areas could include warehouses, shipping/receiving areas, storerooms, courtyards, and/or parking lots.

### A3.4 Health and Safety

All reference analyses and verification testing will follow the safety and health protocols in place for the test facility. This includes maintaining a safe work environment and a current awareness of handling potentially toxic chemicals. Exposure to potentially toxic chemicals will be minimized, personal protective equipment will be worn, and safe laboratory practices will be followed.

**Table 1. General Verification Test Schedule**

| Month | Testing Activities | Data Analysis and Reporting |
|---|---|---|
| 1-3 | • Identify test collaborators/ subcontractors and test facility<br>• Recruit vendors | |
| 3-4 | • Prepare draft TQAP and submit for vendor and peer reviews | |
| 4-5 | • Revise draft TQAP<br>• Finalize and obtain vendor approval of TQAP<br>• Procure necessary standards and reagents<br>• Vendor to set up technology and train technical staff on technology use | |
| 6 | • Conduct verification tests<br>• Conduct reference tests and performance evaluation audit of reference methods<br>• Conduct TSA | • Review and compile test data and records as they become available<br>• Review and summarize verification testing staff observations<br>• Begin preparation of report template |
| 7 | | • Evaluate and analyze data generated during testing<br>• Conduct data quality audits<br>• Complete report template |
| 8 | | • Complete draft reports and submit for vendor and peer review |
| 9 | • Return equipment to vendors | • Revise draft reports and submit final reports for EPA approval |
| 10 | | • Distribute finalized, EPA-approved reports<br>• Post ETV reports and verification statements on ETV Web site |

## A4 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

In performing the verification test, Battelle will follow the technical and QA procedures specified in this protocol and will comply with the data quality requirements in the AMS Center QMP. (1) The objective of this verification test is to evaluate the performance of soil toxicity detecting technologies in their ability to measure the presence of toxins in soil under controlled laboratory conditions. This evaluation will assess the capabilities of the soil toxicity technologies to detect toxins added to a controlled experimental matrix, as well as their ability to detect toxins in "real-world" environmental samples. The evaluation will include a comparison of the soil toxicity technology results to known concentrations of toxins in the test samples that will be confirmed as described in Section B4. Additionally, this verification test will rely upon verification testing staff observations to assess other performance characteristics of the technologies. Below is a discussion of the quality objectives and the criteria for measurement data that have been established to ensure that the test objectives are met.

### A4.1 Quality Objectives

Data quality objectives assure that the data quality, quantity, and type are appropriate to meet the verification test objectives and specify the minimum acceptance criteria for these parameters. Data quality objectives for this verification test include those related to the reference method performance and those related to the soil toxicity detecting technology performance, as well as those related to documenting verification testing staff observations. Data quality objectives for the reference methods (see Section B4) are presented in terms of data quality indicator (DQI) criteria for the critical measurements associated with the reference methods and are listed in Table 2 and discussed in Section A4.2. The reference method data quality relies, in part, on proper sample preparation, proper application of the reference method, and proper maintenance of reference method instrumentation. Battelle will rely on the vendor's data quality objectives for each technology in order to ensure that the technology is performing properly during testing. This will include adhering to each vendor's criteria for calibration and performance of positive and negative control samples. The technology data quality relies on proper operation and maintenance of the technologies and proper sample preparation, as instructed by the vendor. Quantitative data quality objectives for the operator observations have not been defined but are incorporated into documentation requirements and data review, verification, and validation requirements for this verification test.

### A4.2 Criteria for Measurement Data

Table 2 presents the DQIs and criteria for the reference method critical measurements. The reference method measurement quality will be ensured by adhering to these DQI criteria and monitored by following the calibration procedures and frequency recommended in each respective reference method and by including method blank or spiked samples as indicated in each reference method. Additionally, performance test samples will be sent to each laboratory providing reference method analyses prior to analysis of verification test samples. Performance test samples will be standard solutions or standard reference materials containing known quantities of the analytes of interest. Each vendor will provide criteria for the soil toxicity technologies for critical measurements related to calibration standards and recommendations for appropriate positive and negative controls and their critical measurements. The Battelle Quality Manager or his or her designee will perform a TSA at least once during this verification test to review these QA/quality control (QC) requirements. The EPA AMS Center Quality Manager also may conduct an independent TSA, at his or her discretion.

## A5 SPECIAL TRAINING/CERTIFICATION

Documentation of training related to technology testing, field testing, data analysis, and reporting is maintained for all Battelle technical staff in training files at their respective Battelle location. Documentation of the expertise and experience of collaborators and/or subcontractors must be similarly available. The Battelle Quality Manager may verify the presence of appropriate training records prior to the start of testing. If technical staff operate and/or maintain a technology during the verification test, the technology vendor will be required to train those staff prior to the start of testing. Battelle will document this training with a consent form, signed by the vendor, that states which specific technical staff have been trained on their technology.

Battelle technical staff will have a minimum of a bachelor's degree in science/engineering or equivalent work experience.

## A6 DOCUMENTATION AND RECORDS

The records for this verification test will include the TQAP based on this protocol, chain-of-custody (COC) forms, laboratory record books (LRBs), data collection forms, electronic files (both raw data and spreadsheets), and the final verification reports and verification statements. All of these records will be maintained in the Verification Test Coordinator's office or at the test

**Table 2. DQIs and Criteria for Critical Measurements for Reference Method**

| DQI | Method of Assessment | Frequency | Minimum Acceptance Criteria | Corrective Action |
|---|---|---|---|---|
| Bias and Accuracy of Sample Measurements | Initial Calibration—various levels as specified in reference method | As required in reference method | Refer to reference method criteria | Investigate sources of contamination or changes in instrument parameters; perform instrument maintenance as needed; reanalyze fresh standard or sample, or repeat initial calibration. |
| | Calibration Check Sample—single-level continuing check of calibration as specified in reference method | As required in reference method | Refer to reference method criteria | |
| | Method Blank | As required in reference method | Refer to reference method criteria | |
| | Spiked Samples | As required in reference method | Refer to reference method criteria | |
| Completeness | Amount of valid data obtained | Overall number of data points collected for reference method | 90% of overall data points collected should be valid. | If feasible, analyze additional samples to meet the acceptance criterion. |
| Method Representativeness | Performance Test Sample | Once, prior to verification testing | Results within ± 10% of expected value for standard solutions, results within certified limits for standard reference materials | Evaluate reference method performance; perform maintenance or recalibration as required, repeat performance test. If performance test criteria cannot be met, consider alternative reference laboratories. |

facility during the test and will be transferred to the Verification Test Coordinator's office at the conclusion of the verification test. The location (e.g., specific personal computer, server, or media type and storage location) of final versions of the electronic files will be noted in the test records. All Battelle LRBs are stored indefinitely, either by the Verification Test Coordinator or Battelle's Records Management Office. EPA will be notified before disposal of any files. The QA/QC documentation and results of the reference measurements made by the reference laboratory will be submitted to Battelle immediately upon completion of all sample analyses and maintained with the records for this test. Table 3 has further details regarding the data recording practices and responsibilities.

All written records must be in ink. Any corrections to notebook entries, or changes in recorded data, must be made with a single line through the original entry. The correction is then to be entered, initialed, and dated by the person making the correction. In all cases, strict confidentiality of data from each vendor's technology, and strict separation of data from different vendors' technologies, will be maintained. Separate files (including manual records, printouts, and/or electronic data files) will be kept for each technology.

**Table 3. Summary of Data Recording Process**

| Data to Be Recorded | Where Recorded | How Often Recorded | By Whom | Disposition of Data |
|---|---|---|---|---|
| Dates, times, and details of test events, technology maintenance, downtime, etc. | ETV LRBs or data recording forms | Start/end of test procedure, and at each change of a test parameter or change of technology status | Technical staff | Used to organize and check test results; manually incorporated in data spreadsheets as necessary |
| Technology calibration information | ETV LRBs, data recording forms, or electronically | At technology calibration or recalibration | Technical staff or vendor performing the calibration | Incorporated in verification report as necessary |
| Technology readings | Either recorded electronically by the technology and downloaded to an independent computer or storage medium, hard copy data printed by the technology and taped into an ETV LRB, or handwritten records into an ETV LRB or on data sheets | Every sample analysis. | Technical staff | Transferred to or manually entered into spreadsheet for statistical analysis and comparisons |
| Sample preparation and reference method analysis procedures, calibrations, QA, etc. | LRBs, COC, or other data recording forms | Throughout sampling and analysis processes | Technical staff and Reference laboratory | Retained as documentation of reference method performance |
| Reference method results | Electronically from analytical method or documented in handwritten records | Every sample analysis | Reference laboratory | Transferred to or manually entered into spreadsheets for calculation of results, and statistical analysis and comparisons as needed |

# SECTION B
# MEASUREMENT AND DATA ACQUISITION

## B1 EXPERIMENTAL DESIGN

This generic protocol outlines testing for a number of contaminants that are common to site cleanups and known to be toxic to humans, but does not include all toxic compounds or testing in all situations which may be encountered in a site cleanup or evaluation situation. Data generated from verification tests based on this generic protocol are intended to provide one set of objective and quality assured performance data on environmental technologies, to assist users, developers, regulators, and consultants in making informed decisions about purchasing and properly applying these technologies. These technologies do not provide identification or concentration of specific contaminants, but serve as a rapid screening tool to determine whether the soil being tested is toxic. As part of this verification test, the technologies will be subjected to various concentrations of chemicals representing several categories of common contaminants such as commercial solvents, pesticides, persistent pollutants, and metals. At a minimum, the contaminants listed in Table 4 should be evaluated during verification testing. Each contaminant will be added individually to separate aliquots of sand, and the spiked sand will be analyzed by the technologies. Sand is recommended as the matrix for the spiking experiments because it is inert and will minimally retain the contaminants of interest thereby providing an estimate of technology performance in the case where nearly 100% of the contaminant would be bioavailable. Additionally, “real-world” environmental samples of various soil types containing a variety of the contaminants in Table 4 will be analyzed to evaluate the technology performance on samples more representative of those used in practical application of the technologies. These samples are described in Section B1.1.

All of the technologies will be tested in a laboratory. The technologies designed for use in a field location will also be tested at a non-laboratory venue.

The analyses will be performed according to the vendor’s recommended procedures as described in the user’s instructions or manual, or during training provided to the technical staff. Similarly, calibration and maintenance of the technologies will be performed as specified by the vendor. Results from the technologies being verified will be recorded manually by the operator on appropriate data sheets or captured in an electronic data system and then transferred manually or electronically for further data workup. Qualitative operational characteristics of each technology such as ease of use will be assessed through observations made by the Verification Test Coordinator and technical staff throughout the verification test. The results from each technology will be reported individually. No direct comparison will be made between technologies, but each technology will undergo similar testing.

**Table 4. Categories and Contaminants**

| Category | Contaminant |
|---|---|
| Commercial solvents | Trichloroethylene<br>Toluene |
| Carbamate pesticide | Aldicarb |
| Organophosphate pesticide | Dicrotophos |
| Metals | Arsenic<br>Lead<br>Mercury<br>Cadmium |
| Persistent pollutants | Polychlorinated biphenyls (PCBs) (as Aroclor 1254)<br>2,3,7,8- tetrachlorodibenzodioxin (TCDD)<br>Benzo[a]pyrene |

### B1.1 Test Procedures

The verification test for technologies that detect toxicity in soil will focus on a broad range of samples to provide a variety of toxin concentrations. ETV verifications usually include a comparison of the results generated by the technologies being verified with the results of analysis of the same samples using a standard reference method that measures the same endpoint, usually concentration. In the case of this verification test, the standard methods for soil toxicity, such as seed germination and root elongation evaluations, as well as organism-based tests such as earthworm survival, are quite different from those used in rapid soil toxicity technologies. Sensitivity to contaminants will be different among different test species. Therefore, the standard soil toxicity tests would not provide an appropriate benchmark for direct comparison with the results produced by the rapid soil toxicity technologies because of the differing test species. It is well documented that the contaminants used during this verification test are toxic to humans; the objective of the test is to determine each technology's ability to detect this toxicity if the contaminant is present. In lieu of a traditional reference measurement of toxicity, the concentration of each contaminant in the test samples will be confirmed independently by standard laboratory methods.

The first sample type will be performance test samples where individual toxins will be added to sand. Sand is recommended as an inert matrix which will minimally retain the toxins. Use of an inert matrix will eliminate the matrix itself from influencing the lowest detectable concentration of each contaminant and will evaluate technology performance under conditions where the toxin is nearly 100% bioavailable. The sand will be spiked with each contaminant at concentrations ten times screening or remediation goal levels (e.g., EPA Region 9 Superfund Preliminary Remediation Goals) as the highest concentration and will be analyzed in replicate (minimum of three). Subsequent tenfold dilutions will be prepared and analyzed in replicate (minimum of three) until there is no longer a measurable response indicating toxicity (i.e., inhibition as measured by each technology such as a reduction in light output, change in respiration rate, etc.), up to a maximum of five dilutions below the highest concentration. From these data, the lowest concentration at which the toxicity can be detected, or toxicity threshold, will be estimated for each technology with respect to each contaminant. The second sample type will be "real-world" environmental samples and will consist of 5 to 10 soils collected from various cleanup sites or standard reference soils with well documented soil characteristics. These samples will reflect a variety of soil types and will include soils known to contain the contaminants evaluated in this study (individually or in combination with other contaminants) as well as some soils which are known to be free of contaminants, such as American Society of Testing Materials artificial soil or Environmental Resource Associates Semivolatile Blank Soil (Catalog Number 056). The environmental samples will be dried and homogenized prior to use in testing to ensure that sample homogeneity is not a significant factor in technology performance. Because the drying and homogenization process has the potential to affect the concentration of contaminants in the samples, the concentration of contaminants will be measured after the drying and homogenization process has taken place to ensure that the measured concentrations of contaminants in the environmental samples accurately reflect the material used in testing. Contaminants in the environmental samples will be measured using the same reference methods that will be used to confirm the concentration of spiked contaminants in the performance test samples. Appropriate soil characteristics such as total organic carbon, grain size distribution, and pH should also be measured once the environmental samples have been dried and homogenized. Information about the soil characteristics may aid in understanding differences in the various environmental samples that will be tested. To the extent possible, each time verification testing is conducted following this generic protocol, attempts should be made to use the same environmental sites, or at a minimum sites with comparable types of soil and types and quantities of contaminants. The third type of sample will be quality control samples. Quality control samples are discussed further in Section B5.

The technologies will be evaluated for the parameters listed in sections B1.1.1 to B1.1.9.

*B1.1.1 Endpoint*

Each technology produces its own unique endpoint derived from the inhibition data gathered when analyzing various concentrations of contaminants in soil [e.g, median effective concentration causing 50% inhibition ($EC_{50}$) values]. For each technology, the endpoint used for verification testing will be recommended by the vendor.

*B1.1.2 Precision*

Inhibition results (endpoints) specific to each technology from replicates (minimum of three) of each test sample will be evaluated. Relative standard deviation (RSD) of the replicate measurements will be calculated in order to evaluate the precision of the technologies.

*B1.1.3 False Negative Rate*

The false negative rate, or frequency of inhibition similar to the negative control reported when a contaminant is present at toxic concentrations, will be calculated.

*B1.1.4 False Positive Rate*

The false positive rate, or frequency of detectable inhibition reported in unspiked samples, will be calculated.

*B1.1.5 Lowest Detectable Level*

Various contaminants will be added individually to a controlled experimental matrix at multiple concentration levels and analyzed by the participating technologies to assess their ability to detect the toxicity of these contaminants. After analyzing several concentrations of each contaminant, the lowest concentration of each contaminant that causes inhibition statistically greater than that of the negative control will be reported.

*B1.1.6 Matrix Effects*

Various environmental samples representing a variety of soil types and contaminants or mixtures of contaminants will be analyzed. The concentrations of contaminants present in the environmental samples will be measured according to reference methods. The technology's ability to detect contaminants in the environmental samples will be compared with the lowest detectable level of contaminant determined for each technology to assess whether the environmental sample matrix influenced the ability of the technology to detect toxicity.

*B1.1.7 Data Completeness*

Data completeness will be determined as the number of valid measurements out of the total number of measurements taken. The cause of any substantial loss of data will be established from technical staff observations or technology records and noted in the discussion of the data completeness results.

*B1.1.8 Operational Factors*

Operational and sustainability factors such as maintenance needs, calibration frequency, data output, consumables used, ease of use, repair requirements, waste production, and sample throughput will be evaluated based on technical staff and Verification Test Coordinator observations. An LRB or data sheets will be used to document observations. Examples of information to be recorded include the daily status of diagnostic indicators for the technology, use or replacement of any consumables, the effort or cost associated with maintenance or repair, vendor effort (e.g., time on-site) for repair or maintenance, the duration and causes of any technology downtime or data acquisition failure, quantity and hazardous nature of any waste generated, operator observations about technology ease of use, clarity of the vendor's instruction manual, user-friendliness of any needed software, overall convenience of the technologies and accessories/consumables, and the number of samples that could be processed per hour or per day. These observations will be summarized to aid in describing the technology performance in the verification report on each technology.

#### *B1.1.9 Field Portability*

Testing the operation of the technologies in a field setting is a key component of the verification test. Evaluating the performance of each field-portable technology while being used outside the laboratory without the availability of miscellaneous laboratory supplies is important to the buyers and users of these technologies. Technologies will be evaluated in a field setting only if the vendor states that the technology has that capability. For those technologies that are meant to be field-portable, this parameter will be assessed by transporting the technology to a non-laboratory location. In addition to traditional field settings, non-laboratory areas could include warehouses, shipping/receiving areas, storerooms, courtyards, and/or parking lots provided the location meets the criteria that the area is absent of laboratory amenities such as laboratory bench space, power, lighting, storage and refrigeration, etc. as would be the case in a traditional field setting. Ideally all of the samples included in the lab-based tests would be repeated in the field; however, at a minimum one performance test sample or environmental sample that had a strong response in the lab-based tests will be analyzed in triplicate in the field. Results obtained in the field will be compared with the results for the same sample obtained in the laboratory. Technical staff will also record observations related to field portability such as requirements for power, space, and ease of use in and transport to a non-laboratory setting.

## B1.2 Statistical Analysis

The statistical methods and calculations used for evaluation of the quantitative performance parameters are described in the following sections.

### *B.1.2.1 Endpoint*

Each technology produces its own unique endpoint derived from the inhibition data gathered when analyzing various concentrations of contaminants in soil (e.g., $EC_{50}$ values). For each technology, these data will be documented and presented with respect to each contaminant and concentration level using the appropriate endpoint for the technology.

### *B1.2.2 Precision*

The standard deviation ($S$) of the results for the replicate analyses of the same sample will be calculated as follows.

$$S = \left[ \frac{1}{n-1} \sum_{k=1}^{n} \left( M_k - \overline{M} \right)^2 \right]^{1/2} \tag{1}$$

where $n$ is the number of replicate samples, $M_k$ is the endpoint measurement for the $k^{th}$ sample, and $M$ is the average endpoint measurement of the replicate samples. The technology precision for each sample will be reported in terms of RSD, which will be calculated as follows.

$$RSD(\%) = \left| \frac{S}{\overline{M}} \right| \times 100 \tag{2}$$

The RSD values for each analyte at each concentration will be listed in data tables in the verification report; however, verification statements and performance summary tables in the verification report will list the range of RSDs obtained for all concentrations of each contaminant tested.

*B1.2.3 False Negative Rate*

Results will be considered false negative only when a technology is exposed to a contaminant concentration greater than the desired remediation or screening level and the technology does not indicate inhibition greater than the negative control. The rate of false negatives, expressed as a percentage of total samples analyzed for each contaminant, will be calculated by dividing the number of false negative measurements ($M_{fn}$) by the total number of measurements included in verification testing ($M_{total}$).

$$FalseNegative(\%) = \frac{M_{fn}}{M_{total}} \times 100 \qquad (3)$$

*B1.2.4 False Positive Rate*

Results will be considered false positive only when an unspiked sample produces inhibition greater than that of the negative control. The rate of false positives, expressed as a percentage of total samples analyzed for each contaminant, will be calculated by dividing the number of false positive measurements ($M_{fp}$) by the total number of measurements included in verification testing ($M_{total}$).

$$FalsePositive(\%) = \frac{M_{fp}}{M_{total}} \times 100 \qquad (4)$$

*B1.2.5 Lowest Detectable Level*

The lowest detectable level will be the concentration of contaminant in performance test samples (i.e., sand spiked with contaminant) that causes an inhibition greater than the average inhibition of the negative control, which will be calculated using all negative control measurements made during the test. Performance test sample results that are above the average negative control plus the standard deviation about the average will be considered detectable. The lowest performance test sample that results in a detectable inhibition calculated in this manner will be considered the lowest detectable concentration.

*B1.2.6 Matrix Effects*

The technology's ability to detect each contaminant in the environmental samples will be compared with the technology's lowest detectable contaminant level determined by spiking contaminant into an inert matrix (i.e., sand) as described in Section B1.2.5. If the contaminant concentration in the environmental sample (measured using reference methods described in Section B4) is above the lowest detectable level in an inert matrix (as determined in Section B1.2.5), but the technology result for the environmental sample is negative, matrix effects will be considered to have contributed to this false negative response. The number of environmental samples where matrix effects affected results ($M_{matrix}$) out of the total number of environmental samples tested ($M_{total}$) will be reported as a percentage using Equation 5.

$$MatrixEffect(\%) = \frac{M_{matrix}}{M_{total}} \times 100 \qquad (5)$$

*B1.2.7 Data Completeness*

Data completeness will be calculated as the percentage of the total possible data by dividing the number of valid data measurements generated by each technology ($M_{valid}$) by the total number of data measurements included in verification testing ($M_{total}$).

$$Completeness(\%) = \frac{M_{valid}}{M_{total}} \times 100 \qquad (6)$$

The cause of any substantial loss of data will be established from operator observations or technology records and noted in the discussion of the data completeness results.

*B1.2.8 Operational Factors*

There are no statistical calculations applicable to operational factors. Operational factors will be determined based on documented observations of the technical staff and the Verification Test Coordinator.

*B1.2.9 Field Portability*

The results obtained from the measurements made on samples in the laboratory and field setting will be compiled independently for each technology and compared to assess the accuracy of the measurements under the different analysis conditions. Means and standard deviations of the endpoints generated in both locations will be compared and assessed for whether they are statistically different.

## B1.3 Reporting

The data obtained in the verification test will be compiled separately for each vendor's technology, and the data evaluations will be applied to each technology's data set without reference to any other. At no time will data from different vendors' technologies be intercompared or ranked. Following completion of the data evaluations, a draft verification report and verification statement will be prepared for each vendor's technology, stating the verification test procedures and documenting the performance observed. For example, descriptions of the data acquisition procedures, use of vendor-supplied proprietary software, consumables used, repairs and maintenance needed, and the nature of any problems will be presented in the draft report. Each report will briefly describe the ETV program, the AMS Center, and the procedures used in verification testing. The results of the verification test will be stated quantitatively, without comparison to any other technology tested or comment on the acceptability of the technology's performance. Each draft verification report will be submitted for review by the respective technology vendor and by EPA and other peer reviewers. Comments on the draft report will be addressed in revisions of the report. The peer review comments and responses will be tabulated to document the peer review process. The reporting and review process will be conducted according to the requirements of the AMS Center QMP.[1]

## B2 SAMPLING REQUIREMENTS

### B2.1 Sample Collection, Storage, and Shipment

Environmental samples will be collected for use in a verification test following this protocol. As much as possible, samples will be obtained from known contaminated sites using the same sampling techniques that are in place at the site for the site evaluation process. Samples may be collected in bulk and shipped to the test facility in plastic buckets or other suitable containers. Shipments will be via a trackable overnight delivery service to the test facility sample custodian. Samples will be stored refrigerated or frozen as is appropriate for the contaminants expected to be contained in the soil. Environmental samples will be dried and homogenized prior to use in testing to ensure that sample heterogeneity is a minimal factor in testing multiple technologies. Because of the sample handling involved, the environmental samples will be homogenized before concentrations of contaminants are measured using the reference methods. Appropriate soil characteristics such as total organic carbon, grain size distribution, and pH should be measured once the environmental samples have been dried and homogenized. Information about the soil characteristics may aid in understanding differences in the various environmental samples that will be tested.

## B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Sample custody will be documented throughout collection, transport, shipping (if necessary), and analysis using standard COC forms provided by Battelle or supplied by the reference laboratory, as appropriate. Samples transferred within Battelle may be documented in bound sample login LRBs. Each COC form will summarize the samples collected and analyses requested. The COC forms will track sample release from the sampling location to the test facility and/or reference laboratory; or release directly from the test facility to the reference laboratory. Each COC form will be signed by the person relinquishing the samples once that person has verified that the COC form is accurate. The original sample COC forms will accompany the samples; the shipper will keep a copy. Upon receipt at the test facility and/or reference laboratory, COC forms will be signed by the person receiving the samples once that person has verified that all samples identified on the COC forms are present. Any discrepancies will be noted on the form; and the sample receiver will immediately contact the Verification Test Coordinator to report missing, broken, or compromised samples. Copies of all COC forms will be delivered to the Verification Test Coordinator and maintained with the test records.

## B4 LABORATORY REFERENCE METHODS

Table 5 lists the methods that can be used to verify the concentration of contaminants analyzed during verification tests performed following this protocol. Additional methods may be used provided they are appropriate for the contaminant and matrix and are documented in the TQAP for the verification test.

**Table 5. Contaminant Compound Confirmatory Methods**

| Chemical | Method |
|---|---|
| Trichloroethylene, toluene | SW-846 8260B[(4)] |
| Aldicarb | EPA 531.1[(5)] |
| Dicrotophos | SW-846 8141A[(6)] |
| Arsenic, lead, mercury, cadmium | EPA 200.8[(7)] |
| PCBs (as Aroclor 1254) | SW-846 8270C[(8)] |
| 2,3,7,8-TCDD | EPA 1613B[(9)] |
| Benzo[a]pyrene | SW-846 8270C[(8)] |

## B5 QUALITY CONTROL

Steps will be taken to maintain the quality of data collected during verification tests conducted under this protocol. This will include analyzing specific quality control samples (QCS) at a regular frequency by the technologies undergoing verification. The QCSs will include negative controls, positive controls, and calibration checks. Negative control samples, consisting of unspiked experimental matrix, will help ensure that no sources of contamination are introduced in the sample handling and analysis procedures. The positive control and calibration check samples, specified by each vendor, will indicate to the technical staff whether or not the technology is functioning properly. The vendor will provide the approximate endpoint that should result with their technology upon analysis of the positive control and calibration check. QCSs producing results that do not meet the anticipated results specified by the vendor will be reanalyzed and corrective action taken if needed to ensure that test sample results are not affected. Corrective actions may include reanalyzing samples to verify that the technology has been operated properly, conducting maintenance, or recalibrating. Positive and negative controls will be analyzed at a frequency of approximately 5% based on the total number of test samples. Calibration checks will be analyzed according to guidance provided by each technology vendor.

As described in Section B4, the reference laboratory will follow standard reference methods for determining the toxins evaluated during verification tests conducted under this protocol. All reference measurements will be expected to meet the reference method QC requirements (such as those listed in Table 2) or, in absence of specific requirements in the reference method, the reference laboratory's standard requirements for QC samples.

## B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

The equipment used by the test facility and/or reference laboratory will be tested, inspected, and maintained as per the SOPs of the test facility and/or reference laboratory and/or the manufacturer's recommendations so as to meet the performance requirements established in this document. When technical staff operate and maintain technologies undergoing testing, they will follow directions provided by the technology vendor. Otherwise, operation and maintenance of the technologies will be the responsibility of the technology vendor.

## B7 CALIBRATION/VERIFICATION OF TEST PROCEDURES

Systems used for reference analyses will be calibrated as appropriate before any reference samples are analyzed and recalibrated as needed based on the reference methods and/or reference laboratory SOPs.

Technologies undergoing testing will be calibrated initially by the respective technology vendor prior to shipping the technology to the test facility, or during training, and will be recalibrated according to direction from the vendor. Calibration checks will be performed upon direction of the vendor. In the event that recalibration is necessary, the recalibration will be carried out by the technology vendor or by technical staff under the direction of the vendor. All calibrations will be documented as appropriate by the technical staff or vendor.

## B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

All materials, supplies, and consumables will be ordered by the Verification Test Coordinator or designee. Where possible, Battelle will rely on sources of materials and consumables that have been used previously as part of ETV testing without problems. Battelle will also rely on previous experience or recommendations from EPA advisors, stakeholders, test collaborators, subcontractors, or technology vendors. Where possible, materials or supplies will be traceable to the National Institute of Standards and Technology (NIST). Upon receipt of any supplies or consumables, the Verification Test Coordinator or designee will visually inspect and ensure that the materials received are those that were ordered and that there are no visual signs of damage that could compromise the suitability of the materials. Certificates of analysis (COA) or other documentation of analytical purity will be checked for all reagents and standards to ensure suitability for the verification test and will be included with the test files. If damaged, unsuitable, or inappropriate goods are received, they will be returned or disposed of, and arrangements will be made to receive replacement materials.

## B9 NON-DIRECT MEASUREMENTS

No non-direct measurements will be used during this verification test.

## B10 DATA MANAGEMENT

Various types of data will be acquired and recorded electronically or manually by Battelle, vendor, collaborator, and/or subcontractor staff during the verification test. Table 3 summarizes the types of data to be recorded. All maintenance activities, repairs, calibrations, and operator observations relevant to the operation of the technologies will be documented by technical staff in LRBs or on data sheets. Results from the reference methods, including raw data, analyses, and final results, will be compiled by the reference laboratory, preferably in electronic format, and submitted to Battelle at the conclusion of reference method testing.

Records received or generated by any technical staff during the verification test will be reviewed by a Battelle staff member within two weeks of generation or receipt, before the records are used to calculate, evaluate, or report verification results. If a Battelle staff member generated the record, this review will be performed by a Battelle technical staff member involved in the verification test, but not the staff member who originally generated the record. The review will be documented by the person performing the review by adding his/her initials and date to the hard copy of the record being reviewed. In addition, any calculations performed by technical staff will be spot-checked by Battelle QA and/or technical staff to ensure that calculations are performed correctly. Calculations to be checked include any statistical calculations described in this protocol. The data obtained from this verification test will be compiled and reported independently for each technology. Results for technologies from different vendors will not be compared with each other.

Among the QA activities conducted by Battelle QA staff will be an audit of data quality. This audit will consist of a review by the Battelle Quality Manager of at least 10% of the test data. The results of this audit will be compiled in an assessment report. During the course of any such audit, the Battelle Quality Manager will inform the technical staff of any findings and any need for immediate corrective action. If serious data quality problems exist, the Battelle Quality Manager will request that Battelle’s AMS Center Manager issue a stop work order. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem, and will implement any necessary follow-up corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken.

# SECTION C
# ASSESSMENT AND OVERSIGHT

## C1 ASSESSMENTS AND RESPONSE ACTIONS

Every effort will be made in verification tests conducted under this protocol to anticipate and resolve potential problems before the quality of performance is compromised. One of the major objectives of this protocol is to establish mechanisms necessary to ensure this. Internal QC measures described in this protocol, which is peer reviewed by a panel of outside experts, will be implemented by the technical staff and monitored by the Verification Test Coordinator; these QC measures will give information on data quality on a day-to-day basis. The responsibility for interpreting the results of these checks and resolving any potential problems resides with the Verification Test Coordinator. Technical staff have the responsibility to identify problems that could affect data quality or the ability to use the data. Any problems that are identified will be reported to the Verification Test Coordinator, who will work with the Battelle Quality Manager to resolve any issues. Action will be taken to control the problem, identify a solution to the problem, minimize losses and correct data, where possible. Independent of any EPA QA activities, Battelle will be responsible for ensuring that the audits described in the following sections are conducted as part of this verification test.

### C1.1 Performance Evaluation Audits

A performance evaluation (PE) audit will be conducted to assess the quality of the reference method measurements made in this verification test. The PE audit of the reference methods will be performed by supplying each reference method a blind sample or standard reference material containing the toxins of interest. The PE audit samples will be analyzed in the same manner as all other samples, and the analytical results for the PE audit samples will be compared with the nominal concentration or certified value. The target criterion for this PE audit is agreement of the analytical result within 25% of the nominal concentration [by percent difference (PD)] or within 25% of the certified value (by PD). If the PE audit results do not meet the tolerances shown, they will be repeated. If the outlying results persist, a change in reference instrument and a repeat of the PE audit may be considered. This audit will be performed once prior to the start of the test and will be the responsibility of the Verification Test Coordinator or designee.

### C1.2 Technical Systems Audits

The Battelle Quality Manager or designee will perform a TSA at least once during verification tests conducted under this protocol. The purpose of this audit is to ensure that the verification test is being performed in accordance with the AMS Center QMP[(1)], this protocol, published reference methods, and any SOPs used by the reference laboratory. In the TSA, the Battelle Quality Manager, or a designee, may review the reference methods used, compare actual test procedures to those specified or referenced in this protocol, and review data acquisition and handling procedures. In the TSA, the Battelle Quality Manager will tour the test facility, observe sample collection if appropriate, inspect documentation of sample COC, and review technology-specific records. He or she will also check standard certifications and technology data acquisition procedures and may confer with the technology vendors, reference laboratory, and technical staff. The Battelle Quality Manager may also visit the reference laboratory to review procedures and adherence to this plan and applicable SOPs. A TSA report will be prepared, including a statement of findings and the actions taken to address any adverse findings. The EPA AMS Center Quality Manager will receive a copy of Battelle's TSA report. At EPA's discretion, EPA QA staff may also conduct an independent on-site TSA during the verification test. The TSA findings will be communicated to technical staff at the time of the audit and documented in a TSA report.

### C1.3 Data Quality Audits

The Battelle Quality Manager or designee will audit at least 10% of the verification data acquired in the verification test. The Battelle Quality Manager will trace the data from initial acquisition, through reduction and statistical comparisons, to final reporting. All calculations performed on the data undergoing the audit will be checked.

### C1.4 QA/QC Reporting

Each assessment and audit will be documented and submitted in accordance with Sections 3.3.4 and 3.3.5 of the AMS Center QMP.[1] The results of the TSA will be submitted to EPA. Assessment reports will include the following:

- Identification of any adverse findings or potential problems
- Response to adverse findings or potential problems
- Recommendations for resolving problems
- Confirmation that solutions have been implemented and are effective
- Citation of any noteworthy practices that may be of use to others.

## C2 REPORTS TO MANAGEMENT

The Battelle Quality Manager, during the course of any assessment or audit, will identify to the technical staff performing experimental activities any immediate corrective action that should be taken. If serious quality problems exist, the Battelle Quality Manager is authorized to request that Battelle's AMS Center Manager issue a stop work order. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem and will implement any necessary follow-up corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken. This protocol, any TQAPs based on this protocol, and final verification reports are reviewed by EPA AMS Center QA staff and EPA AMS Center program management staff. Upon final review and approval, both documents will be posted on the ETV Web site (www.epa.gov/etv).

# SECTION D
# DATA VALIDATION AND USABILITY

## D1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

The key data review requirements for the verification test are stated in Section B10 of this protocol. In general, the data review requirements specify that the data generated during this test will be reviewed by a Battelle technical staff member within two weeks of generation of the data. The reviewer will be familiar with the technical aspects of the verification test, but will not be the person who generated the data. This process will serve both as the data review and the data verification and will ensure that the data have been recorded, transmitted, and processed properly. Furthermore, this process will ensure that the soil toxicity detecting technology data and the reference method data were collected under appropriate testing conditions and that the reference method data meet the specifications of the reference method.

The data validation requirements for this test involve an assessment of the data quality relative to the DQIs and audit acceptance criteria specified for this test. The DQIs listed in Section B5 will be used to validate the quality of the data. The QA audits described within Section C of this document, including the PE audit and audit of data quality, are designed to validate the quality of the data.

## D2 VALIDATION AND VERIFICATION METHODS

Data verification is conducted as part of the data review, as described in Section B10 of this protocol. A visual inspection of handwritten data will be conducted to ensure that all entries were properly recorded or transcribed and that any erroneous entries were properly noted (i.e., single line through the entry with an explanation of the error and the initials of the recorder and date of entry). Electronic data from the technologies and other instruments used during the test will be inspected to ensure proper transfer from the datalogging system. Data manually incorporated into spreadsheets for use in calculations will be checked against handwritten data to ensure that transcription errors have not occurred. All calculations used to transform the data will be reviewed to ensure the accuracy and the appropriateness of the calculations. Calculations performed manually will be reviewed and repeated using a handheld calculator or commercial software (e.g., Excel). Calculations performed using standard commercial office software (e.g., Excel) will be reviewed by inspecting the equations used in calculations and verifying selected calculations by handheld calculator. Calculations performed using specialized commercial software (i.e., for analytical instrumentation) will be reviewed by inspecting and, when feasible, verifying by handheld calculator or standard commercial office software.

To ensure that the data generated from this test meet the goals of the test, a number of data validation procedures will be performed. Section C of this protocol describes the validation safeguards employed for this verification test. Data validation and verification efforts include the completion of QC activities and the performance of TSA and PE audits as described in Section C. The data from this test will be evaluated relative to the measurement DQIs described in Section B5, and the PE audit acceptance criteria given in Section C1.1 of this protocol. Data failing to meet these criteria will be flagged in the data set and not used for evaluation of the technologies, unless these deviations are accompanied by descriptions that adequately demonstrate that data quality was not compromised.

An audit of data quality will be conducted by the Battelle Quality Manager to ensure that data review, verification, and validation procedures were completed and to assure the overall data quality. The schedule for completing TSA, PE and audits of data quality are included in Table 1.

## D3 RECONCILIATION WITH USER REQUIREMENTS

The purpose of a verification test performed following this protocol is to evaluate the performance of commercial technologies for detecting toxicity in soil. In part, this evaluation will include comparisons of the results from the technologies to results from established reference methods. To meet the requirements of the user community, the data obtained in such a verification test should include thorough documentation of the performance of the technologies during the verification test. The data review, verification, and validation procedures described above will ensure that verification test data meet these requirements and are accurately presented in the verification reports generated from the test and that data not meeting these requirements are appropriately flagged and discussed in the verification reports. Additionally, all data generated using reference methods that are used to evaluate technology results during the verification test should meet the QA requirements of the reference methods.

This generic verification protocol and any resulting ETV verification report(s) generated following procedures described in this protocol will be reviewed by participating technology vendors, ETV AMS Center staff, test collaborators, EPA, and external expert peer reviewers. These reviews will ensure that this protocol, verification test(s) of technologies for detecting toxicity in soil, and the resulting report(s) meet the needs of potential users and regulators. The final report(s) will be submitted to EPA in Microsoft Word and in 508 compliant Adobe Portable Document Format (pdf) and subsequently posted on the ETV Web site.

# SECTION E

# REFERENCES

## E1 REFERENCES

1. "Quality Management Plan (QMP) for the ETV Advanced Monitoring Systems Center" (AMS Center QMP), U.S. EPA Environmental Technology Verification Program, prepared by Battelle, Columbus, Ohio, Version 6.0, November 2005.

2. "Environmental Technology Verification Program Quality Management Plan" (EPA ETV QMP), December 2002, EPA/600/R-03/021.

3. "ECO Update: Catalogue of Standard Toxicity Tests for Ecological Risk Assessment," EPA Office of Solid Waste and Emergency Response, March 1994, Publication 9345.0-05I.

4. SW-846 Method 8260B, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometery (GC/MS)," Revision 2, 1996.

5. EPA Method 531.1, "Measurement of n-Methylcarbamoyloximes and n-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Post Column Derivatization," Revision 3.1, 1995.

6. SW-846 Method 8141A, "Organophosphorous Compounds by Gas Chromatography: Capillary Column Technique," Revision 1, September 1994.

7. EPA Method 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively-Coupled Plasma Mass Spectrometry," Revision 5.4, 1994.

8. SW-846 Method 8270C, "Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)," Revision 3, 1996.

9. EPA Method 1613, "Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS," Revision B, 1994.